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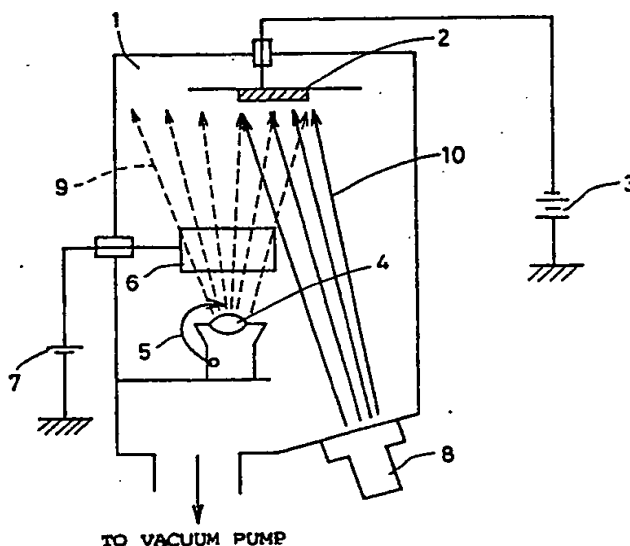
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54 **Surface treatment process.**

57 A surface treatment process for forming a thin film on the surface of a substrate thereby to obtain a hard layer, by forming a metal vapor atmosphere around the substrate to inject a metal or nonmetal ion into the substrate while ionizing the metal vapor.



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SURFACE TREATMENT PROCESS

The present invention relates to a surface treatment process for forming a thin film on the surface of a substrate thereby to obtain a hard layer.

Description of the Prior Art

5 In order to obtain hard layers on the surfaces of substrates, there have been proposed a PVD (physical vapor deposition) process such as an ion plating process and a CVD (chemical vapor deposition) process employing chemical reaction under a high temperature. These processes are
10 adapted to form hard layers of carbide, nitride, oxide, boride or the like of several microns in thickness on the surfaces of substrates. The hard layers thus formed are dispersed in thickness, and hence the said processes cannot be applied to usage directed to accuracy.

15 The CVD process is adapted to form a thin film under a considerably high temperature, whereby adhesion is obtained finely to some extent between the substrate and the surface-coating film. In this process, however, an embrittled layer is formed between the substrate and the
20 surface-coating film, thereby to reduce strength of the substrate. On the other hand, the ion plating process is

adapted to form a hard layer under a temperature relatively low in comparison with the CVD process, whereby no sufficient adhesion is obtained between the substrate and the surface-coating film.

5 Proposed in recent years is a process of injecting a nonmetal ion into a substrate as a surface treatment process for forming a hard layer. However, such process cannot be applied to all types of substrates, and application thereof is limited to those of materials which
10 react with a nonmetal ion to be hardened. For example, the surface layer is hardened by injecting a nitrogen ion into an Fe-alloy, whereas no surface layer is hardened by injecting the nitrogen ion into a Co-alloy or Ni-alloy.

 In order to overcome the aforementioned problem, the
15 inventors have proposed an improved surface treatment process in Japanese patent application No. 64507/1982 (patent laying-open gazette No. 181864/1983) filed on April 16, 1982. This surface treatment process is
20 characterized in that the nitrogen ion is injected into a substrate in a metal vapor atmosphere provided around the substrate to be treated. When, for example, Ti is
 employed as an atmospheric metal in this process, Ti is also injected with the nitrogen ion into the surface of the substrate. Thus, Ti and the nitrogen ion partially
25 react with each other to generate strong nuclei of TiN on

the surface, thereby to facilitate hardening of the surface. However, there is yet room for improvement in this process. Namely, Ti and N in the surface-coating film are preferably provided in the form of TiN coupled substantially in the ratio of 1:1 in order to improve 5 adhesion between the substrate and the surface-coating film and increase hardness of the surface-coating film. However, the rate of Ti, which serves as the atmospheric metal, is inevitably increased in the aforementioned 10 process combining vapor deposition and ion injection.

SUMMARY OF THE INVENTION

Accordingly, an object of the present invention is to provide a surface treatment process which develops and improves the process disclosed in Japanese patent 15 application No. 64507/1982 to further improve adhesion between the substrate and the surface-coating film, and which can appropriately adjust the ratio of concentration of the atmospheric metal and the injected ion.

The present invention provides a surface treatment 20 process which is characterized in that a metal vapor atmosphere is formed around a treated substrate thereby to inject a metal or nonmetal ion into the substrate with the metal vapor being ionized.

According to the present invention, metal particles 25 in the ionized metal vapor reach the treated substrate

under high energy to firmly adhere to the substrate.
Further, the metal particles in the metal vapor are
injected into the surface of the substrate with the ion to
be injected. The evaporation metal and the injected ion
5 are thus knocked on the substrate so that no clear
interface is present between the substrate and the
surface-coating film, thereby to improve adhesion of the
same. Preferably, the injected ion is accelerated by an
ion source to strike the surface of the treated substrate
10 thereby to activate the said surface, and hence adhesion
of the substrate and the surface-coating film is further
facilitated. Further, since the metal particles in the
metal vapor are ionized, reactivity thereof is increased
to form a hard compound such as TiN, thereby to facilitate
15 hardening of the surface.

In addition, conditions such as the amount of
evaporation of the metal, the degree of ionization of the
metal vapor, the amount of an acceleration voltage applied
to the treated substrate, the amount of an acceleration
20 voltage to the injected ion, the amount of the injected
ion and the temperature of the substrate may be
appropriately set in order to optimumly adjust the ratio
of concentration of the metal particles in the metal vapor
forming the surface-coating film to the injected ion.
25 Thus, when, for example, Ti is employed as the evaporation

metal and N is employed as the injected ion, Ti and N can be provided in the ratio of 1:1.

Preferably the evaporation metal is formed by a substance or two or more substances selected from a group of Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W, Si, Al and B and the injected metal or nonmetal ion is formed by a substance or two or more substances selected from a group of N_2^+ , N^+ , O_2^+ , C^+ , Si^+ and B^+ . Typical combinations for obtaining excellent hard layers are, for example, Ti employed as the evaporation metal and N_2^+ employed as the injected ion, Al employed as the evaporation metal and O_2^+ employed as the injected ion, Ti employed as the evaporation metal and C^+ employed as the injected ion and B employed as the evaporation metal and N_2^+ employed as the injected ion.

Ionization of the metal vapor is effectively performed by, for example, radio-frequency discharge, DC arc discharge and thermoelectronic emission. Further, an HCD (hollow cathode discharge) gun may be employed in order to obtain the metal vapor and ionize the same by electrons of the HCD gun. Any metal may be readily ionized by the radio-frequency discharge and the thermoelectronic emission. Ionization by the DC arc discharge is efficiently performed, whereas metals to be ionized are limited. For example, the DC arc discharge is effective in the case where Ti is employed as the

evaporation metal. The HCD gun can ionize any metal in excellent efficiency. The aforementioned ionization means are not necessarily employed separately, and, for example, the thermoelectronic emission and the DC arc discharge can
5 be combined or the HCD gun can be combined with the radio-frequency discharge. Such combination further facilitates ionization of the evaporation metal. Further, an acceleration voltage may be applied to the treated substrate in order to increase the incident speed of the
10 ionized metal particles, so that the ionized metal particles forcibly strike the surface of the substrate thereby to further improve adhesion of the surface-coating film.

A compound of the evaporation metal and the injected
15 ion may be formed and coated on the treated substrate. For example, in the case where Ti is employed as the evaporation metal and N is employed as the injected ion, hardening of the surface is improved by forming and coating TiN on the treated substrate. Although it is not
20 particularly necessary for the object of the present invention, mass spectrometry may be performed on extracted and accelerated ions to extract a specific type of ion to introduce the same into an evaporation chamber.

The present invention is particularly effectively
25 applied to the case of forming and coating carbide,

nitride, oxide or boride on the substrate in order to increase abrasion resistance and corrosion resistance thereof. The present invention is remarkably effective on a treated substrate requiring adhesion of the surface-coating film such as a cutting tool.

The above and other objects, features, aspects and advantages of the present invention will become more apparent from the following detailed description of the present invention when taken in conjunction with the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 illustrates an example of a device employed for carrying out the present invention;

Fig. 2 illustrates another example of a device for carrying out the present invention;

Fig. 3 illustrates still another example of a device for carrying out the present invention; and

Fig. 4 illustrates a further example of a device for carrying out the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Example 1

A surface treatment device as shown in Fig. 1 was employed to form a hard layer on the surface of a substrate. A substrate material 2 is arranged within a vacuum evaporation chamber 1. The substrate material 2 is

connected with a substrate accelerating power source 3, which is adapted to apply a voltage to the same. A metal 4 is contained in a crucible arranged in the vacuum evaporation chamber 1. The metal 4 is evaporated by 5 electron beams 5, to travel along paths 9. Arranged in the travel paths 9 is an ionization electrode 6 which is connected to an ionization power source 7, and the metal vapor is ionized by the ionization electrode 6. Further, an ion generated by an ion source 8 is accelerated at its 10 extracted portion, to travel in the evaporation chamber 1 along paths 10. Thus, the ionized metal vapor and the ion emitted from the ion source 8 enter the surface of the substrate 2 to form a hard layer.

In this example, Ti was employed as the metal 4 and 15 N_2^+ was employed as the injected ion. The substrate 2 was a small-diameter drill ($\phi 0.90$ mm) made of cemented carbides. A sample A-1 was prepared with the atmospheric pressure of 1.3×10^{-4} Pa, acceleration voltage of 0.8 KV applied to the substrate 2, acceleration voltage of 40 KV 20 applied to the injected ion and voltage of 50 V at the ionization power source 7. The degree of ionization of the metal vapor was 75 %.

Samples as comparison materials were prepared in the following manner: In a sample B-1, N_2^+ ion injection was 25 performed while evaporating Ti, and the evaporation metal

Ti was not ionized. Only N_2^+ ion injection was performed in a sample C-1 while TiN was coated by the CVD process on a sample D-1. TiN was coated by the ion plating process on a sample E-1, while no treatment was made on a sample F-1.

The said samples were subjected to cutting tests under the following conditions:

Workpiece: three-ply printed substrate (G10)

Revolution of Drill: 80,000 r.p.m.

Feed of Drill: 0.05 mm/rev.

Table 1 shows the hit counts to the duration of lives of the respective samples.

Table 1

<u>Sample</u>	<u>Hit Count</u>
A-1 (sample obtained in accordance with the present invention)	11,100
B-1 (prior art)	6,900
C-1 (prior art)	5,500
D-1 (prior art)	1,800
E-1 (prior art)	4,300
F-1 (untreated)	3,500

Example 2

A surface treatment device as shown in Fig. 2 was employed to form a hard layer on a substrate 2. Reference numerals 1 to 5 and 8 to 10 indicate components substantially identical to those in Fig. 1, and explanation thereof is omitted. In this example, an RF electrode 6a for ionization is arranged in a vacuum evaporation chamber 1 in order to ionize the evaporation metal. The electrode 6a is connected with a matching unit 11 and a radio-frequency power source 7a.

A cutting insert of M10 cemented carbide (SNG432) was employed as the substrate 2 and Al was employed as the evaporation metal 4, while O_2^+ was employed as the injected ion. A test was made with the atmospheric pressure of 2.5×10^{-3} Pa, the acceleration voltage of 0.2 KV applied to the substrate 2, the acceleration voltage of 25 KV applied to the injected ion and the electric power of 500 W at the radio-frequency power source 7a. The degree of ionization of the evaporation metal was 18 %. A sample A-2 was thus obtained.

The following samples were obtained as comparison materials. In a sample B-2, O_2^+ ion injection was made while evaporating Al without ionization. Only O_2 ion injection was performed in a sample C-2, while Al_2O_3 was

coated by the CVD process on a sample D-2. Al_2O_3 was coated by the ion plating process on a sample E-2, while no treatment was made on a sample F-2.

These samples were subjected to cutting tests under
5 the following conditions:

Workpiece: SCM435(H)

Cutting Speed: 150 m/min.

Feed: 0.36 mm/rev.

Cutting Depth: 1.5 mm

10 The lives of the respective samples were measured when the flank wear V_B reached 0.20 mm to obtain results tabulated in Table 2. It is obvious from Table 2 that the sample A-2 obtained by the surface treatment process according to the present invention is further superior to
15 the other samples.

Table 2

<u>Sample</u>	<u>Life</u>
A-2 (sample obtained in accordance with the present invention)	25 min.
B-2 (prior art)	14 min.
C-2 (prior art)	1 min.
D-2 (prior art)	15 min.
E-2 (prior art)	3 min.
F-2 (untreated)	5 sec.

Example 3

A surface treatment device as shown in Fig. 3 was employed to form a hard layer on the surface of a substrate. Reference numerals 1 to 5 and 8 to 10 indicate components substantially identical to those in Fig. 1. In this example, ionization of evaporation metal 4 is performed by a thermoelectronic emission filament 6b connected to a filament power source 7b.

A sample A-3 was prepared by a substrate 2 formed by SNG432 of high speed steel, the evaporation metal 4 of Ti and injected ion of C^+ . The atmospheric pressure was 5×10^{-4} Pa, the acceleration voltage applied to the substrate 2 was 1.2 KV, the acceleration voltage to the

injected ion was 60 KV, the voltage at the filament power source 7b was 12 V and the degree of ionization of the metal vapor was 63 %.

The following samples were obtained as comparison materials: In a sample B-3, C^+ ion injection was performed while evaporating Ti without ionization. Only C^+ ion injection was performed in a sample C-3 while TiC was coated by the CVD process on a sample D-3. TiC was coated by the ion plating process on a sample E-3, while no treatment was made in a sample F-3.

These samples were subjected to cutting tests under the following conditions:

Workpiece: 18-8 stainless steel

Cutting Speed: 40 m/min.

Feed: 0.1 mm/rev.

Cutting Depth: 1.0 mm

The lives of the respective samples were measured to reach the duration to obtain results tabulated in Table 3.

Table 3

<u>Sample</u>	<u>Life</u>
A-3 (sample obtained in accordance with the present invention)	20 min.
B-3 (prior art)	13 min.
C-3 (prior art)	9 min.
D-3 (prior art)	8 min.
E-3 (prior art)	5 min.
F-3 (untreated)	3 min.

Example 4

A surface treatment device as shown in Fig. 4 was employed to form a hard layer on the surface of a substrate. Reference numerals 1 to 5 and 8 to 10 as shown in Fig. 4 indicate components substantially identical to those in Fig. 1. In this example, an HCD gun 6c connected to an HCD gun power source 7c was adapted to evaporate molten metal 4. Further, ionization of the metal vapor was performed by electrons of the HCD gun 6c.

10 A sample A-4 was formed by a substrate 2 prepared by a cutting insert of cemented carbide K10, the evaporation metal of B and the injected ion of N_2^+ . The atmospheric

pressure was 3×10^{-4} Pa, the acceleration voltage applied to the substrate 2 was 2.0 KV, the acceleration voltage to the injected ion was 100 KV and the degree of ionization of the metal vapor was 82 %.

5 The following samples were formed as comparison materials: In a sample B-4, N_2^+ ion injection was performed while evaporating B without ionization. Only N_2^+ ion injection was performed in a sample C-4 while BN was coated by the CVD process on a sample D-4. BN was
10 coated by the ion plating process on a sample E-4, while no treatment was made on a sample F-4.

These samples were subjected to cutting tests under the following conditions:

Workpiece: FCD45
15 Cutting Speed: 300 m/min.
Feed: 0.1 mm/rev.
Cutting Depth: 0.4 mm

The lives of the respective samples were measured to reach the duration to obtain results tabulated in Table 4.

Table 4

<u>Sample</u>	<u>Life</u>
A-4 (sample obtained in accordance with the present invention)	12 min.
B-4 (prior art)	5 min.
C-4 (prior art)	3 min.
D-4 (prior art)	1.5 min.
E-4 (prior art)	1 min.
F-4 (untreated)	30 sec.

Although the present invention has been described and illustrated in detail, it is clearly understood that the same is by way of illustration and example only and is not to be taken by way of limitation, the spirit and scope of
5 the present invention being limited only by the terms of the appended claims.

WHAT IS CLAIMED IS:

1. A surface treatment process of forming a metal vapor atmosphere around a substrate to be treated thereby to inject a metal or nonmetal ion into said substrate while ionizing said metal vapor.
2. A surface treatment process in accordance with claim 1, wherein said substrate to be treated is a cutting tool.
3. A surface treatment process in accordance with claim 1, wherein said evaporation metal is made of a substance or two or more substances selected from a group of Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W, Si, Al and B.
4. A surface treatment process in accordance with claim 1, wherein said injected metal or nonmetal ion is made of a substance or two or more substances selected from a group of N_2^+ , N^+ , O_2^+ , O^+ , C^+ , Si^+ and B^+ .
5. A surface treatment process in accordance with claim 1, wherein said ionization of said metal vapor is performed by high-frequency discharge.

6. A surface treatment process in accordance with claim 1, wherein said ionization of said metal vapor is performed by DC arc discharge.

7. A surface treatment process in accordance with claim 1, wherein said ionization of said metal vapor is performed by thermoion emission.

8. A surface treatment process in accordance with claim 1, wherein said metal vapor is obtained by an HCD gun, said ionization of said metal vapor being performed by electrons of said HCD gun.

9. A surface treatment process in accordance with claim 1, wherein an acceleration voltage is applied to said substrate to be treated.

10. A surface treatment process in accordance with claim 1, wherein a compound of said evaporation metal and said injected ion is formed and coated on said substrate to be treated.

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FIG. 1

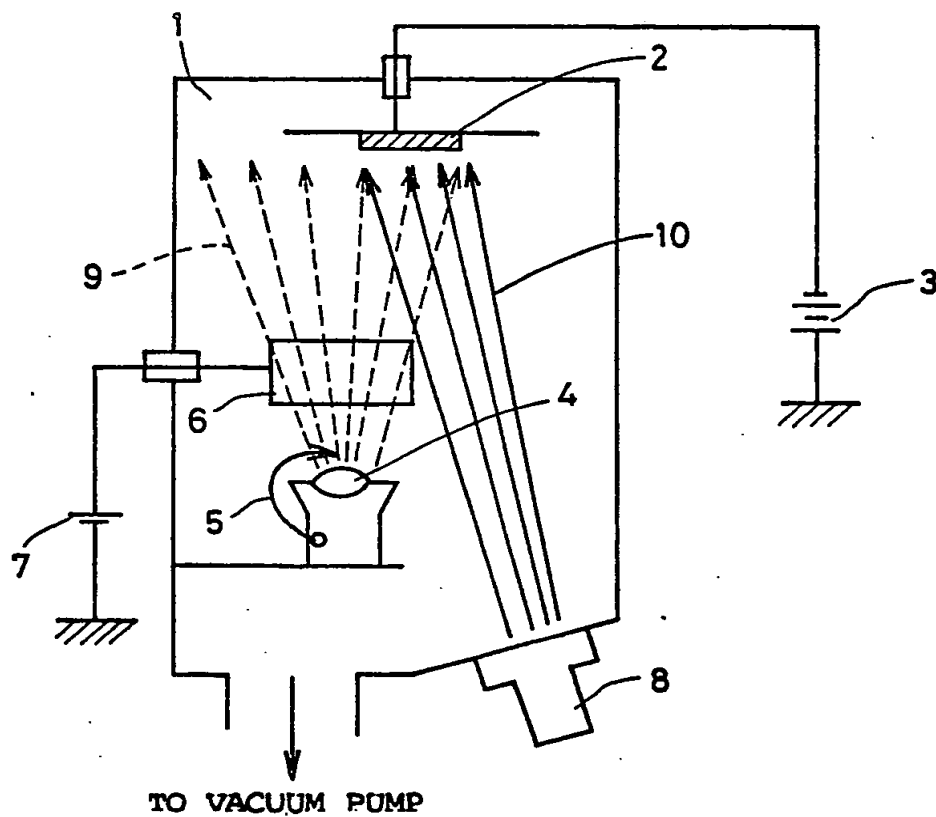
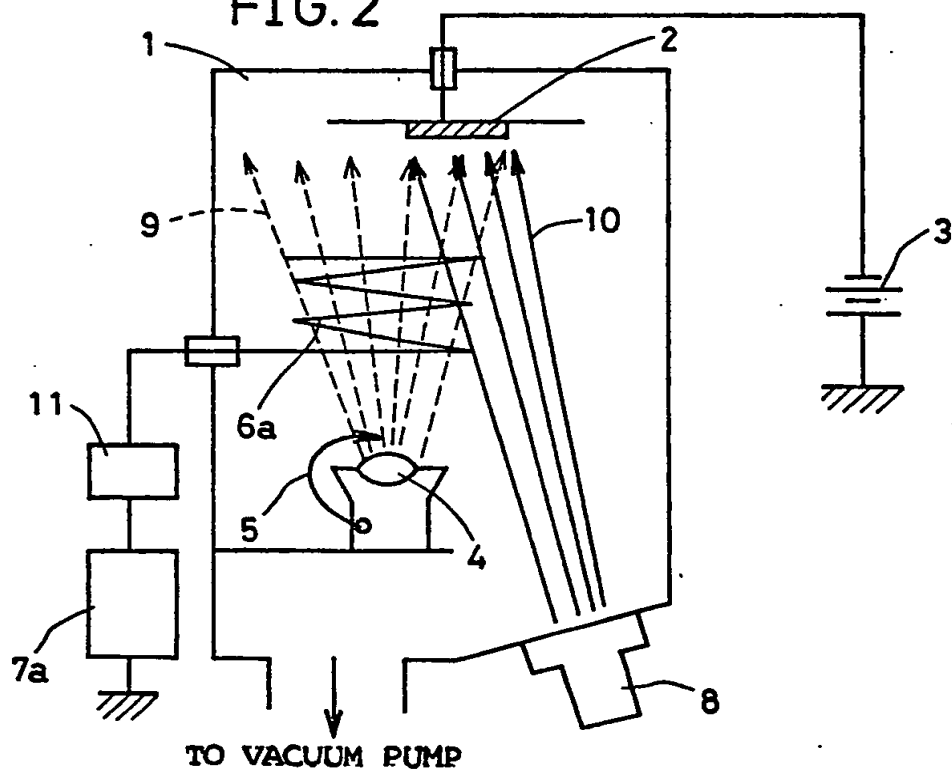


FIG. 2



2/2

FIG. 3

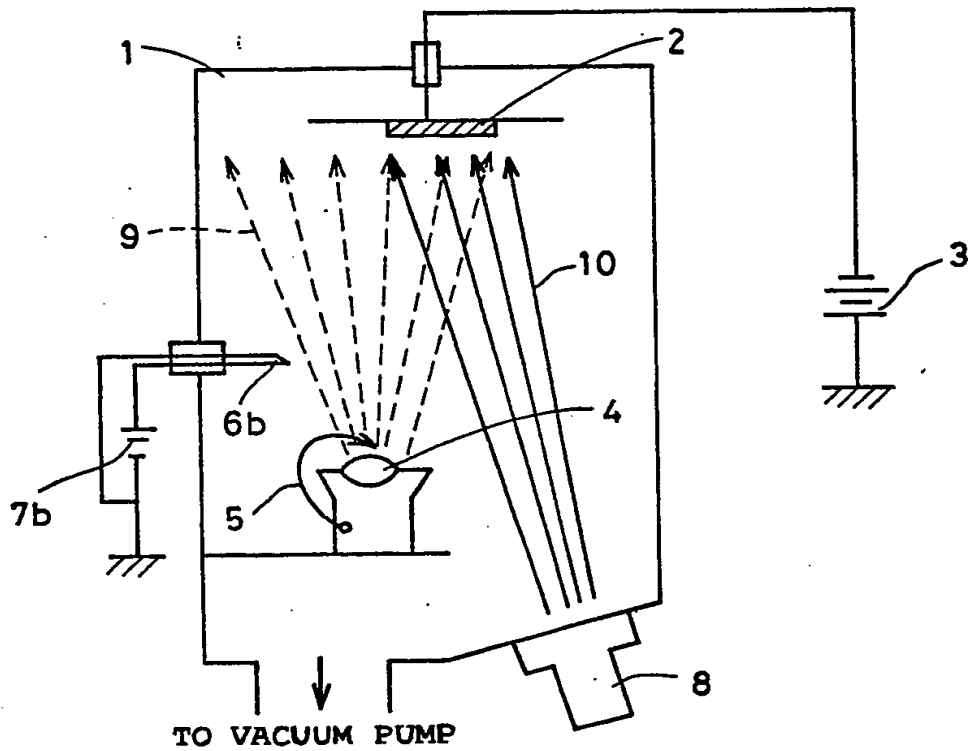
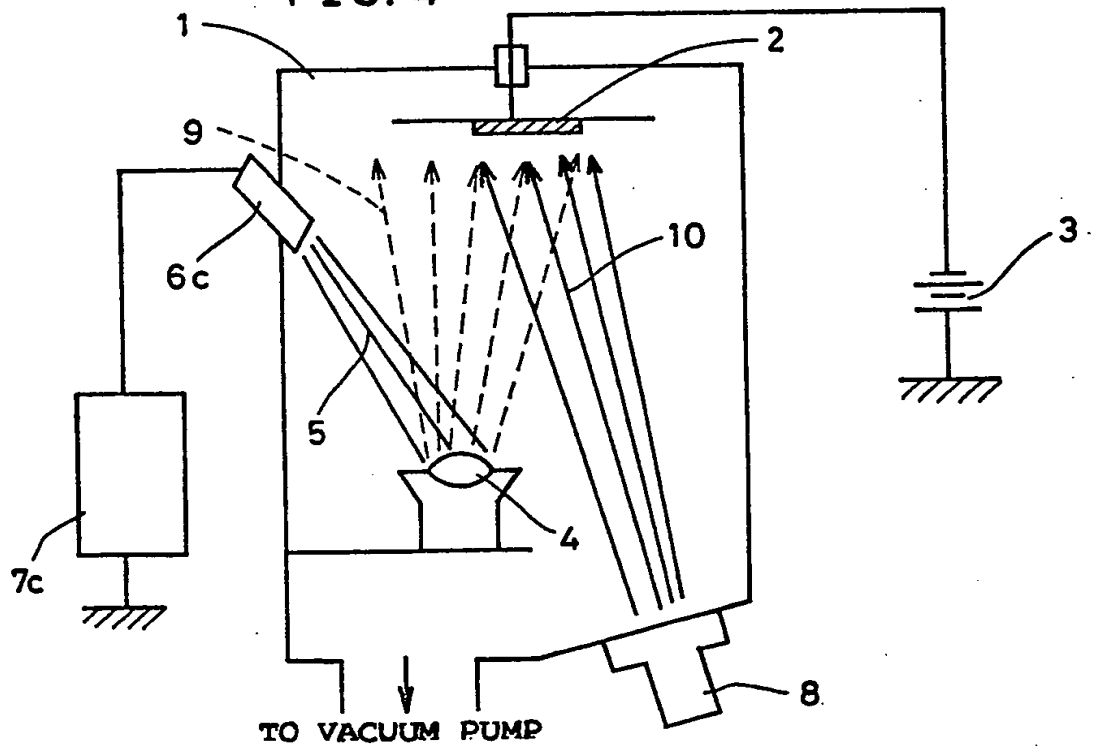


FIG. 4





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EUROPEAN SEARCH REPORT

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Application number

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The present search report has been drawn up for all claims			
Place of search VIENNA		Date of completion of the search 03-09-1985	Examiner SLAMA
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p>			



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A	US - A - 4 407 712 (W.F. HENSHAW et al.) * Fig.; claims *	1,8	
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A	<p>PATENT ABSTRACTS OF JAPAN, unexamined applications, C field, vol. 4, no. 155, October 29, 1980</p> <p>THE PATENT OFFICE JAPANESE GOVERNMENT page 97 C 29</p> <p>* Kokai-no. 55-100 975 (CITIZEN TOKEI K.K.) *</p> <p>----</p>	1,8	
The present search report has been drawn up for all claims			TECHNICAL FIELDS SEARCHED (Int. Cl. 4)
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